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RESEARCH MEMORANDUM

PREPARATION AND PROPERTIES OF CONCENTRATED BORON-

HYDROCARBON SLURRY FUELS

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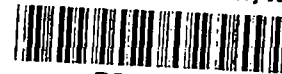(OFFICER AUTHORIZED TO CHANGE)

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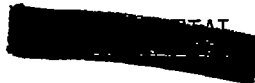# NATIONAL ADVISORY COMMITTEE
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RESEARCH MEMORANDUM

PREPARATION AND PROPERTIES OF CONCENTRATED BORON-HYDROCARBON SLURRY FUELS

By Irving A. Goodman and Virginia O. Fenn

SUMMARY

Fluid slurry fuels were prepared, containing 50 to 60 percent by weight of boron powder of 1-micron average particle size in JP-4 and JP-5 fuels. The boron-hydrocarbon mixtures were fluidized by means of glycerol sorbitan laurate, a surface-active agent, and stabilized with small amounts of aluminum octoate, a gelling agent. Duplicate batches prepared with identical materials on the same day had closely similar properties.

The useful life of a slurry is approximated by the age at which the settled portion of the slurry can no longer be readily redispersed. Satisfactory stability for up to 6 months was observed for several slurries, but since some of these slurries were still in a satisfactory physical state at the time of publication of this report, the maximum total useful life was not determined.

The viscosity at low rates of shear varied with the age of the slurry, but did not follow the same pattern for all slurries. Some slurries reached a maximum viscosity very rapidly, then dropped off gradually from this peak; some did not reach a viscosity peak for 1 or 2 weeks before leveling off or dropping; others remained at a fairly constant viscosity level for prolonged periods; while still others exhibited somewhat erratic viscosity behavior.

The apparent viscosity of the slurry at low rate of shear increased with increasing boron and aluminum octoate concentrations, but decreased with increasing concentration of glycerol sorbitan laurate. The JP-5 slurries seemed to exhibit a slightly higher viscosity than the corresponding JP-4 slurries, probably because of the higher density of the JP-5.

For a given composition, the viscosities of slurries prepared with boron powders containing traces of acid were considerably lower than for those prepared with essentially neutral powder. The stability of
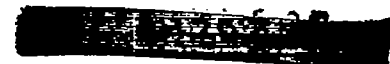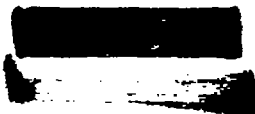
slurries prepared with the acidic powders was generally poorer, particularly in the case of one boron sample which had a relatively high moisture content.

INTRODUCTION

As part of a program involving the preparation of concentrated, stable, fluid slurries of solids of high heat content in hydrocarbons, and the evaluation of these slurries as fuels for ram-jet and afterburner applications, an investigation was undertaken to prepare and study the properties of such slurries containing boron powder as the solid component. The attributes of a desirable slurry for ram-jet fuel applications are outlined in an earlier report (ref. 1). The investigation of boron as a component of such a fuel was undertaken because of its relatively high heat of combustion, making it attractive on both a weight and a volume basis for long-range flight application (ref. 2).

Previous reports (refs. 3 to 6) describe the preparation and the combustion of boron slurries containing 30 to 50 percent by weight of boron powder. The boron used in most of this earlier work was supplied from a variety of sources and had appreciable differences in purity and particle size. At a later date, a single source of high-grade boron powder became available in sufficient quantities to supply the needs of an extensive fuel-evaluation program. It then became desirable to undertake a study of the numerous factors affecting the preparation and the properties of slurries containing this material. Since the need in the combustion field is in the direction of maximum boron concentration, the study was limited to boron concentrations of 50 percent by weight or greater.

When mixed with 50 percent of MIL-F-5624A, grade JP-4 fuel, or similar hydrocarbon fuel, this boron powder yielded an almost completely dry, pasty mass, entirely unsatisfactory for practical use as a fuel. The addition of small quantities of certain surface-active agents had the effect of fluidizing the mixture very markedly. The rapid settling of the powder resulting from this fluidization necessitated the addition of small amounts of a gelling agent in order to control the viscosity and to improve the stability of the slurry. With this combination of additives, it was also observed that the settling which did occur as the slurry aged was frequently in the form of a soft, easily redispersed layer.

The present work is a study of the effects of varying the concentration of the specific surface-active agent, the gelling agent, and the boron powder on the physical characteristics of the slurry, as indicated by apparent viscosity measurements at a low rate of shear. Also included in this study are: (1) the development of a suitable method of slurry preparation, (2) a check on the reproducibility of this method, (3) an

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examination of the variations resulting from a change in the lot or drum of boron powder used, (4) an investigation into the effects of changing the hydrocarbon component from grade JP-4 to JP-5 fuel, and (5) a presentation of viscosity data obtained by different methods.

MATERIALS

Boron. - The boron powder used was a commercial product prepared by the thermal reduction of boric oxide with magnesium. Analysis of a large number of samples from various drums showed that the purity ranged from 87 to 91 percent free boron. Among the known impurities were magnesium, oxides of boron, moisture, and as was later detected in some of the samples, traces of acid. Average particle sizes ranged from approximately 0.6 to 1.4 microns, as determined with the air permeability method by the Fisher Sub-Sieve Sizer. Analyses of samples from the six different containers used in this investigation are given in table I. Electron micrographs of a sample of the powder are shown in figure 1. The major portion of the work was done with two 20-pound drums (1 and 2) of powder having similar properties.

As may be noted from table I, the major single difference in properties among the six boron samples examined appears to be acidity. This was measured as the pH of the water solution obtained by stirring 1 gram of boron powder into 100 milliliters of distilled water, then filtering. The acid, which is present in very small amounts, results from a step in the boron production process involving flotation in an acidic solution (ref. 7). Three of the six samples examined gave pH values appreciably below the average of 5.35 to 6.0 for the distilled water used. One of these also had a moisture content considerably higher than the others, as indicated by weight loss after heating for 1 hour at 105° C.

Additives. - The gelling agent used was aluminum octoate ("fast gelling"), an aluminum chloride precipitated soap produced from 2-ethylhexoic acid, by the Witco Chemical Company. To avoid variations which might be introduced when changing from one lot to another, enough material for the entire investigation was withdrawn from a 50-pound container and stored in a tightly closed gallon can. Small amounts of this material were withdrawn from time to time and placed in a small bottle for use in current slurry preparation. Analysis of this material is presented in table II.

The surface-active agent used throughout this investigation was glycerol sorbitan laurate, produced by the Atlas Powder Company and designated by the trade name, G-672. At room temperature, this material is an amber-colored, viscous liquid with a density of 1.00 to 1.05 grams per milliliter and viscosity of 1800 to 2000 centipoises. As in the case of the gelling agent, all the material used in the investigation was part of a single lot.

Hydrocarbon medium. - With the exception of a small group of samples prepared with JP-4 fuel for purposes of comparison, JP-5 fuel was used throughout the investigation. Prior to use in this study, both fuels had been percolated through columns packed with activated alumina. Analyses and properties of these fuels before treatment, except as noted, are presented in table III.

APPARATUS

Mixing procedure. - It was extremely difficult or impossible to prepare most of the slurries by manual stirring with a spatula or by the use of any conventional laboratory mechanical mixer. The powder was apparently not readily wetted and the resulting mixture was a dry pasty mass. A high-speed commercially-produced mixing unit, especially designed for difficult dispersing problems, was found to be satisfactory for intensive mixing. A 1/2-horsepower motor drives a 1-inch stainless-steel mixing head on a 1/2-inch shaft, as illustrated in figure 2. The speed can be continuously varied from zero to 8000 rpm by means of a transformer.

Viscometers. - There are a large number of viscometers on the market, each with its own modifications, advantages, and disadvantages, designed for use with materials of the type treated in this report. The three instruments used in this work yield three different kinds of data and are fairly typical of the type of instrument they represent. It should be emphasized, however, that practically every instrument in this field differs in some respect from every other and that these three instruments were used because they were available and well known.

(1) A model LVF Brookfield Synchrolectric rotational viscometer was used to follow changes in apparent viscosity at low rates of shear ($< 10 \ sec^{-1}$) with time. Because of the speed and simplicity of operation, most of the data reported herein were obtained with this viscometer. The instrument, its method of operation, and precision are described fully in reference 1.

(2) The Severs Extrusion Rheometer may be used to determine variations in shearing stress with rate of shear for a slurry, based on changes in flow rates through a tubular orifice with stepwise variations in applied air pressure. This instrument is particularly suited to operation at high rates of shear. It is described and illustrated in reference 1, which also explains how apparent viscosity values may be computed from shearing stress - rate of shear data.

(3) The Stormer rotational viscometer (fig. 3) is a modified version of the standard instrument distributed by the Central Scientific Company. It operates at fairly low rates of shear and allows for

stepwise variation in shear rate by the successive addition of weights. The modification consists of the replacement of bob and cup supplied by the manufacturer. The cylindrical cup is made of brass and has a diameter of 1.1395 inches and a length of about $2\frac{5}{8}$ inches. The cylindrical bob is solid stainless steel with a diameter of 1.0225 inches and a length of 1.577 inches. The clearance between centered bob and cup is 0.0585 inch or 1.49 millimeters.

EXPERIMENTAL

Slurry Composition Variables

As indicated before, each slurry consists of a mixture of four different ingredients, including (1) boron powder, (2) aluminum octoate, (3) glycerol sorbitan laurate, and (4) hydrocarbon fuel. It was therefore desirable to investigate the effect of varying the concentration and, in some cases, the properties, of each of these components on the properties of the slurry.

A minimum of 50 percent boron is required to realize appreciable advantages as a fuel with respect to theoretical heat release. Preliminary experiments dictated the practical range of concentration of the additives based on the fluidity of the resultant slurries. The hydrocarbon concentration was always the difference between 100 percent and the sum of the boron and additive concentrations.

The following table indicates the ranges over which these concentrations were varied to make up the samples studied in this investigation. All numbers indicate weight percent of a given constituent.

Boron	Aluminum octoate	Glycerol sorbitan laurate	JP-4	JP-5
50	0.2 to 0.4 .3 .3 .3	1.4 to 2.0 .5 to 4.0 1.6 1.6	48.1	47.6 to 48.4 45.7 to 49.2 48.1
55	0.1 to 0.3 .1 to .3	1.4 to 2.0 1.4 to 2.0	42.7 to 43.4	42.7 to 43.5
60	0 to 0.2	1.6 to 2.0		37.8 to 38.4
50 to 64	0	2.0	34 to 48	

Two sets of slurries, identical except for JP fuel used, were prepared to provide a comparison of slurry properties with JP-4 and JP-5 as carrier fuels. Three samples were made with JP-4 and three with JP-5. The results of the viscosity measurements for each set of slurries were used to evaluate the reproducibility of the results of this investigation.

Another variable studied was the acidity of the boron powder. The effect of this variable on the viscosity and the useful life of slurries was investigated by making slurries with four samples of powder which had different acidities as measured by the pH of an aqueous extract of the powder. The composition of all slurries in this particular set was 50 percent boron, 1.6 percent glycerol sorbitan laurate, 0.3 percent aluminum octoate, and 48.1 percent JP-5.

As the result of a preliminary series of studies involving principally the order of addition of the various slurry components, the following procedure for slurry preparation was developed as the most satisfactory, and was used throughout this program:

The gelling agent is weighed carefully to 0.1 gram and added to a clean, tin-plated, paint can (1-pint capacity). A weighed amount of hydrocarbon is poured over the gelling agent and the mixture is stirred for 1 minute with the mixing unit to insure uniformity. The surface-active agent is weighed directly into the mixture, which is stirred again, as described. The boron powder is weighed into a separate container. It is then added gradually to the mixture, accompanied by occasional stirring with a spatula, until the mixture becomes too thick to stir manually. It is then stirred with the mixing unit until it becomes more fluid. Again boron is added until a thick paste results, and the mixing unit is used as before. This procedure is repeated until all the boron powder has been added. Unwetted boron powder frequently collects in the mixing head, partially clogging the slots and holes. After all the boron powder has been added, the boron remaining in the head is scraped out and added to the slurry. The slurry is further mixed at high speed, until it is smooth and free from lumps. The total slurry weight was 400 grams for the 50 percent and 500 grams for the 55- and 60-percent-boron slurries.

Measurement of Viscosities of Slurries

Brookfield viscometer. - Brookfield apparent viscosity determinations were made for each sample on the morning following the day of preparation and repeated approximately weekly thereafter until the sample was discarded. The sample was discarded when the settled portion of the slurry became so hard and difficult to redisperse that it was deemed of little practical value to extend the data any further. Prior to each viscosity determination, the physical appearance of the slurry

was noted qualitatively, and the slurry was carefully stirred with a spatula until it appeared to be homogeneous. All the viscosities were measured without the use of the spindle guard.

Severs Rheometer. - Data were obtained on this instrument only for selected samples, since there was insufficient material in a given sample for both Brookfield apparent viscosity-time data and Severs Rheometer studies. The data reported herein were obtained 1 day after the slurry was prepared. The orifice selected for a given experiment was the smallest (in diameter) through which steady flow could be maintained. The weight of slurry flowing through the orifice in a measured time was recorded for each of several applied air pressures, starting with the lowest pressure which would just initiate flow. These weights were converted to volumes on the basis of computed slurry densities, and the volume rates of flow, along with pressure values and orifice constants, were used to calculate shear stresses, shear rates, and apparent viscosities.

Stormer viscometer. - Measurements were made 1 day after the samples were prepared. The procedure used in obtaining data on this instrument may be generalized as follows:

A sufficient quantity of slurry is poured into the cup, so that when the cup is raised to where the bottom is just 1.0 centimeter from the bottom of the bob, the top of the bob is immersed in the slurry. A thermostatically controlled heater maintains the desired temperature of approximately 30° C in the water bath which houses and supports the cup. Starting with a weight on the holder sufficient to cause rotation of the bob at the rate of at least 2 rpm, the time required for the bob to rotate a whole number of revolutions (2, 5, 10, 20, 50, or 100) in a convenient length of time (no more than 2 minutes) is measured by a stop watch. This operation is repeated as rapidly as possible with successive additions of 100-gram weights until a rotational speed of approximately 20 rps is attained or a maximum total weight of 3100 grams has been applied, whichever occurs first. The reverse procedure is then started immediately by removing weights, 100 grams at a time, and measuring rotational speeds for each applied weight, as before. Data are plotted as revolutions per second against weight applied, and usually give two curves, the so-called "up curve" connecting points obtained as weights were added, and "down curves", as weights were removed.

RESULTS AND DISCUSSION

The results showing the effect of ingredient concentration on Brookfield apparent viscosity of the slurries are presented by means of viscosity measurements taken 1 day after the sample was prepared. These data are shown as curves, with Brookfield apparent viscosity plotted against concentration of a single ingredient with all other factors held

constant. The change in Brookfield apparent viscosity with time is pre-
sented to compare the slurries on the basis of rate of gel breakdown.
Viscosity is plotted against time, and the results are discussed as they
appear to be related to the composition of the slurry sample. Data for
change of apparent viscosity with rate of shear are presented to illus-
trate the behavior that might be exhibited by slurry fuels when subjected
to varying rates of shear as they flow through the component parts of the
fuel system. Change of viscosity with rate of shear is discussed in re-
lation to slurry composition.

Reproducibility of Stabilized Boron Slurries

Successive batches of slurries of identical composition made from
the same lots of ingredients had closely similar Brookfield apparent
viscosities. Table IV shows the results obtained for two sets of three
slurries each, one set prepared using JP-4, the other using JP-5 fuel
as the hydrocarbon medium. All the slurries contained 50 percent boron,
1.6 percent glycerol sorbitan laurate, and 0.3 percent aluminum octoate,
and were prepared on the same day, using the same lot of each ingredient.
The Brookfield apparent viscosities of the JP-4 slurries 1 day after
preparation ranged from 6120 to 6700 centipoises, the mean viscosity was
6495 centipoises, and the average deviation from the mean was 250 centi-
poises. The viscosities of the JP-5 slurries 1 day after preparation
ranged from 11,500 to 13,900 centipoises, the mean viscosity was 12,600
centipoises, and the average deviation from the mean was 865 centipoises.

Somewhat larger variations in results were sometimes noted among
identical slurries prepared according to the same procedure on different
days. This increase in variation is due possibly to changes in humidity
causing increased moisture absorption which could significantly affect
the gelling properties of the aluminum octoate. These effects were not
further investigated.

Evaluation of Useful Life of Slurries

An attempt was made to estimate the length of time during which a
given slurry may be considered to be useful as a fuel. None of the
slurries prepared were permanently stable in the sense that there was
no settling. The settled portion which did form, however, was of such
a consistency that it could be readily redispersed. This settled portion
usually became harder and harder as time passed until it was extremely
difficult to redisperse. Viscosity determinations were discontinued at
this point, and the slurry age at which this is done was arbitrarily
considered to be the useful life.

Satisfactory stability for as long as 6 months was observed, but the maximum useful life is not known, since some of the most stable slurries had not yet been discarded at the time of publication of this report. In general, the higher the peak viscosity of a slurry, the longer is its useful life.

Factors Affecting Physical Properties of Boron Slurries

Concentration of boron. - Five slurries were prepared, each containing 2.0 percent glycerol sorbitan laurate (no aluminum octoate) with boron concentrations of 50, 55, 58, 60, and 64 percent by weight in JP-4 fuel. In the absence of aluminum octoate, it is possible to prepare slurries with greater than 60-percent-boron concentration, but the stability of such slurries is very poor. The Brookfield apparent viscosities 1 day after preparation are plotted in figure 4, and show an exponential increase with increase in boron concentration. A similar plot is presented for slurries containing 50, 55, and 60 percent boron in JP-5 fuel, with 2.0 percent glycerol sorbitan laurate and 0.2 percent aluminum octoate. The viscosities are lower for the corresponding boron slurries containing no aluminum octoate because of the hydrocarbon-gelling action of the aluminum octoate. An exponential increase in viscosity with increasing boron concentration is again indicated.

Concentration of additives. - In figure 5 is shown the effect of increasing aluminum octoate concentration on the Brookfield apparent viscosity of 50, 55, and 60-percent-boron slurries in JP-5 fuel, for a given concentration of glycerol sorbitan laurate. A definite increase in viscosity is noted for each successive increase in aluminum octoate concentration except for the lowest concentration of aluminum octoate, 0.1 percent. A concentration of 0.1 percent is apparently insufficient to demonstrate a thickening effect, as indicated by the fact that the 60-percent-boron slurry containing 0.1 percent aluminum octoate has a Brookfield apparent viscosity very close to that of the corresponding slurry containing no aluminum octoate.

In figure 6, the effects of varying glycerol sorbitan laurate concentration, within a relatively narrow range, on the Brookfield apparent viscosities of 50- and 60-percent-boron slurries in JP-5 fuel, with aluminum octoate concentration as a parameter are presented. Since two different drums of boron were used in preparing duplicate samples, the plotted points are designated according to the drum of boron from which the sample was prepared. Over the range of glycerol sorbitan laurate concentration investigated, the effects are not very pronounced, nor do they appear to be consistent. Actually, considering the fact that all the samples were not prepared on the same day, whatever variations are indicated could have been caused by day-to-day changes in certain external factors such as humidity.

The gross effects caused by relatively large changes in glycerol sorbitan laurate concentration may be noted in figure 7, in which are plotted viscosity data for 50-percent-boron slurries in JP-5 fuel, with 0.3 percent aluminum octoate, and glycerol sorbitan laurate concentrations ranging from 0.5 to 4.0 percent by weight. The boron powder used for this set of samples came from drum 6.

These data show that there is a significant effect on viscosity when the glycerol sorbitan laurate concentration is changed in larger increments, at least for the specific boron and aluminum octoate concentrations studied. Successive decreases in Brookfield apparent viscosity are observed as the glycerol sorbitan laurate concentration is increased by increments of 0.5 to 1 percent. It may also be noted that the portion of the curve between the limits of 1.4 and 2.0 percent is relatively flat compared with the steep portions on either side of these limits. This difference in slope confirms the results shown in figure 6, which indicate the relatively insignificant effect of glycerol sorbitan laurate variation within these limits.

Type of hydrocarbon fuel. - The Brookfield apparent viscosity data for three identical slurries prepared with JP-4 fuel are presented in table IV for comparison along with similar data obtained with a set of three identical slurries prepared with JP-5 fuel. While the results are not conclusive, certain tentative observations may be noted.

At the end of the first day, the viscosity of the JP-5 slurries had reached a definitely higher level than that of the JP-4 slurries. At the end of 1 week, the JP-4 slurries had increased in viscosity to almost twice their original values and were very close to the viscosities of the JP-5 slurries, which increased only slightly or not at all. After 16 days, the viscosities of the JP-4 slurries had decreased fairly rapidly almost to their original values, while those of the JP-5 samples changed only very slightly. The rates of increase and decrease were apparently appreciably greater during the first 2 or 3 weeks. Viscosity readings were taken again at the end of 4 weeks and then approximately weekly for 3 more weeks. A generally decreasing viscosity pattern was observed during this period. The viscosity of the JP-5 slurries remained generally higher than the corresponding JP-4 slurries, although the rate of viscosity decrease appeared to be somewhat greater for the JP-5 slurries. The higher viscosity of the JP-5 slurries is no doubt due, at least in part, to the higher density of JP-5 which results in a corresponding lower volume of liquid component in the slurry for a given weight.

From the preceding observations and from some unpublished information, it appears that either JP-4 or JP-5 fuel (or similar types) may be used to prepare slurries, with little or no modification required in the concentrations of additives used.

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Boron acidity. - Viscosity-time plots are presented in figure 8 for four slurries having identical composition except for the boron sample used. Samples from drums 2 to 5 were used to prepare the slurries. These curves indicate that the acidity of the boron sample has a profound effect on the viscosity of a given slurry. All three slurries prepared with the boron samples of greater acidity showed low viscosities within hours after their preparation, while the sample of lesser acidity gave a slurry of considerably higher viscosity.

A sample from drum 5, which showed an appreciably higher moisture content (percent volatile) in addition to its higher acidity, produced the slurry with the shortest life. Samples from drums 3 and 4, on the other hand, which had low moisture content and rather high acidity, yielded slurries with relatively good stability in spite of their low viscosities. A sample from drum 2, having low moisture content and lower acidity, yielded the slurry with the longest life and highest viscosity.

It would appear, then, that in order to insure uniform results in slurry preparation, the acidity of the boron powder should be controlled at a constant level by the supplier. In addition, powder containers should be kept sealed until just before use, in order to minimize moisture absorption which may be appreciable for boron powder (ref. 8) and could seriously affect the slurry properties.

Aging. - In figure 9, several typical plots of Brookfield apparent viscosity against time after preparation, for 50-, 55-, and 60-percent-boron slurries in JP-4 and JP-5 fuel containing various concentrations of aluminum octoate and glycerol sorbitan laurate, may be seen. The changes in viscosity with time may be attributed to several possible causes.

It has been demonstrated (ref. 9) that hydrocarbon gels made with low concentrations of metal soap show a gradual structure breakdown with time. The rate of breakdown appears to be a function of such factors as soap concentration, soap composition, hydrocarbon structure, and temperature. This breakdown is manifested as a decrease in apparent viscosity. It is probable, too, that the surface-active agent does not exert its full effect instantaneously, but rather over an extended period of time. This gradual wetting action should also be reflected in a gradually decreasing viscosity until the wetting action is essentially completed.

For several of the slurries studied, as indicated in figure 9, an increase in viscosity was observed during the first week, followed by a period of leveling off or gradual decrease in viscosity. This initial period of increasing viscosity may be explained on the basis of a continuous rather than instantaneous gelation process, which is not always complete by the end of the first or second day.

Some of the slurries apparently reached their peak viscosity very soon after they were prepared. This peak was generally followed by a gradual decrease in viscosity. In a few instances, notably for the 60-percent slurries, the viscosity remained at a relatively constant level. This may be due to the fact that for the higher boron concentrations, the principal contribution to viscosity was made by the solids rather than the gelling agent, which was present in very low concentrations. Even in the absence of any aluminum octoate, a 60-percent-boron slurry with 1.6 percent glycerol sorbitan laurate had a viscosity of about 6000 centipoises, as shown in figure 9(c), but the stability was very poor.

The apparently erratic viscosity behavior observed in some cases (e.g., fig. 9(a)) may be the result of inadequate mixing before determining the viscosity, or the formation of lumps during the aging period.

Supplementary Viscosity Data

Severs Extrusion Rheometer. - In figure 10 is shown the variation of shearing stress with rate of shear for three representative slurry compositions measured in the Severs Rheometer. Except for one of the slurries at relatively low rate of shear, straight-line relations appear to exist for the range of shear rates covered.

When the apparent viscosity is computed from the ratio of shearing stress to rate of shear and plotted against the reciprocal rate of shear, the curves shown in figure 11 are obtained. Residual viscosities (ref. 1) may be obtained by extrapolation of the curves to infinite rate of shear. The plot obtained for the 55-percent-boron slurry indicates virtually no change in apparent viscosity over the range investigated, although the viscosity level is considerably lower than the corresponding Brookfield apparent viscosity. The 50-percent-boron slurry containing 0.3 percent aluminum octoate had a much higher Brookfield apparent viscosity than the 50-percent slurry containing 0.2 percent aluminum octoate, but showed a very rapid drop in apparent viscosity with increasing rate of shear. Although the Brookfield apparent viscosities of the 50- and 55-percent-boron slurries containing 0.2 percent aluminum octoate were very similar, the apparent viscosity of the 50-percent slurry at high rates of shear was appreciably lower. For all three cases, the apparent viscosities computed from the Severs Rheometer data at high rates of shear were much lower than the corresponding Brookfield apparent viscosities.

Stormer rotational viscometer. - Three sets of data are plotted in figure 12 for the 50-, 55-, and 60-percent-boron slurries containing various concentrations of aluminum octoate. These data were obtained by use of the Stormer rotational viscometer as described. It may be noted that, for the 50- and 55-percent slurries, the area of the loop

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appears to increase with increasing aluminum octoate concentration, possibly indicating increased thixotropy. The 60-percent-boron slurries do not show any noticeable difference in loop area for the various aluminum octoate concentrations. Although it is impossible at this time to draw any significant conclusions from these data regarding the rheology of these systems, the data are presented as being typical of the patterns obtained with the instrument for such systems.

SUMMARY OF RESULTS

In a study of boron-hydrocarbon slurry fuels prepared with 1-micron boron powder, the following results were obtained:

1. Successive batches of boron slurry made on the same day from the same lots of ingredients had similar Brookfield apparent viscosities.

2. Satisfactory stability for up to 6 months was observed for some of the slurries, but since these slurries were still in a stable condition at the time of publication of this report, the maximum total useful life could not be reported.

3. Increasing the concentration of boron in a slurry from 50 to 60 percent by weight caused exponential increases in the initial Brookfield apparent viscosity.

4. Increasing the aluminum octoate concentration of the slurry in the range 0.1 to 0.4 percent by weight produced marked increases in Brookfield apparent viscosity.

5. Small increases in glycerol sorbitan laurate concentration in the range 1.4 to 2.0 percent by weight did not cause appreciable viscosity changes, but over a range of 0.5 to 4.0 percent by weight, definite decreases in viscosity were observed as the concentration was increased.

6. Slurries prepared with JP-5 fuel appeared to reach their peak Brookfield apparent viscosity much sooner than those prepared with JP-4 fuel, and maintained a slightly higher viscosity level. The differences in viscosity may be due to the difference in hydrocarbon density, which resulted in a smaller JP-5 volume for a given weight, compared with JP-4.

7. For a given slurry composition, boron powders possessing greater acidity, as measured by the pH of a water extract, yielded slurries having much lower Brookfield apparent viscosities. Higher moisture (volatile) content of the powder appeared to shorten the slurry life.

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8. The Brookfield apparent viscosities varied with time for all except the 60-percent-boron slurries, but did not show the same variation pattern for all slurries.

9. The apparent viscosities of boron slurries decreased greatly at high rates of shear compared with those obtained at very low rates of shear.

10. Rotational viscometer data on some of the slurries seemed to indicate varying degrees of thixotropy dependent on aluminum octoate concentration, but these conclusions are only tentative.

CONCLUDING REMARKS

This experimental investigation of the preparation and properties of boron-hydrocarbon slurry fuels has demonstrated that stable fluid slurries containing 50 to 60 percent by weight of boron can be prepared. Good quality control of the ingredients used is necessary, however, to obtain reproducible properties in the slurry samples produced. Particular attention should be given (1) to the acidity and moisture content of the boron powder, (2) constant quality of (dry) hydrocarbon used for carrier, and (3) selection and handling of the aluminum octoate gel agent. By observing proper precautions, a satisfactory slurry can be prepared by appropriate adjustment of the concentration of the various constituents.

Lewis Flight Propulsion Laboratory
    National Advisory Committee for Aeronautics
        Cleveland, Ohio, June 28, 1954

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      Rep. Dec. 1, 1953-Mar. 1, 1954, American Potash & Chem. Corp., Mar.
      15, 1954. (Contract AF 33(616)-488.)

8. Mazza, Harold, and Sawyer, Dwight L.: Status of Research on the Large
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      Rep. June 1-Sept. 1, 1953, American Potash & Chem. Corp., Sept.
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TABLE I. - ANALYSIS OF BORON POWDER

Drum	Average particle diameter of powder, microns	Weight loss at 105° C, percent	Free boron, percent	Acidity of water extract, pH
1	0.92	---	87.4	---
2	.90	0.4	89.9	5.2
3	1.06	.42	89.9	4.05
4	1.05	.47	89.9	4.55
5	1.4	2.4	88.8	4.2
6	1.10	.25	89.6	5.50

TABLE II. - ANALYSIS OF ALUMINUM OCTOATE

Analysis	Percent by weight
Total ash	17.1
Water-soluble salts	.4
Free fatty acid (acetone-soluble)	.9
Moisture	1.2

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TABLE III. - PROPERTIES OF HYDROCARBON FUELS

Properties	MIL-F-7914, grade JP-5	MIL-F-5624A, grade JP-4
Distillation range, $^{\circ}$F		
Initial boiling point	370	140
Percent evaporated		
5	388	199
10	397	222
20	406	248
30	414	268
40	424	286
50	433	300
60	441	325
70	452	348
80	465	382
90	487	427
95	506	459
Final boiling point	534	488
Residue, percent	1.2	1
Aromatics,		
Silica gel, percent by volume	10.4	9.8
Specific gravity, 60/60° F	.816	.768
Density[a] at 68° F, g/ml	.8115	.7676
Hydrogen-carbon ratio	.162	.169
Heat of combustion, Btu/lb	18,625	18,675
Aniline point, $^{\circ}$F	153.1	134.6

[a]Obtained on material passed through activated alumina.

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TABLE IV. - COMPARISON OF BROOKFIELD APPARENT VISCOSITIES OF

SLURRIES PREPARED WITH JP-4 AND JP-5 FUELS

[Slurry composition: 50 percent boron; 1.6 percent glycerol sorbitan laurate; 0.3 percent aluminum octoate; 48.1 percent hydrocarbon.]

Hydrocarbon fuel	Brookfield apparent viscosity (centipoises) after						
	1 day	7 days	16 days	28 days	37 days	44 days	49 days
MIL-F-5624A, grade JP-4	6,120	11,900	6,700	6,130	5870	5820	5140
	6,700	12,000	7,320	6,760	5930	5550	5150
	6,660	11,100	7,980	6,860	5750	5830	5400
MIL-F-7914, grade JP-5	11,500	13,500	12,000	9,970	9590	8660	7840
	12,400	13,900	11,700	10,400	7060	6850	6080
	13,900	13,600	14,700	11,500	8740	7320	6060

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Figure 1. - Electron micrographs of boron powder.

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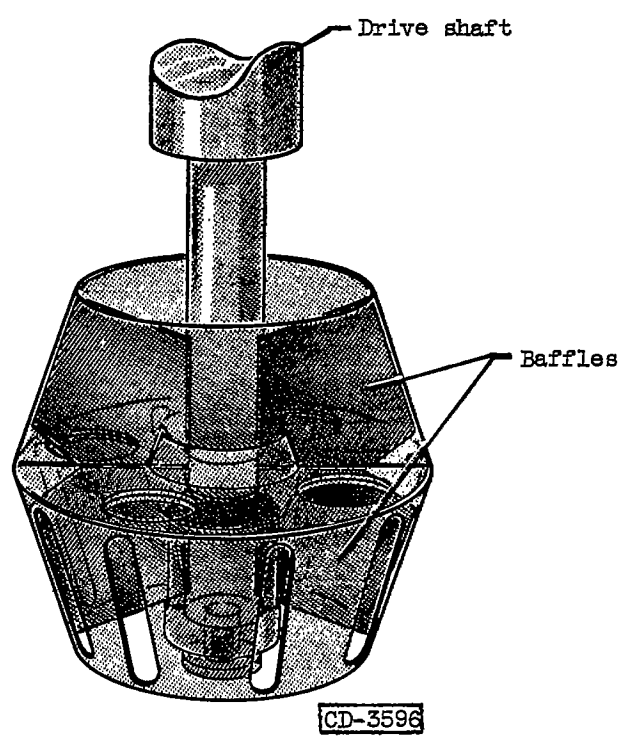
Figure 2. - Mixing unit.
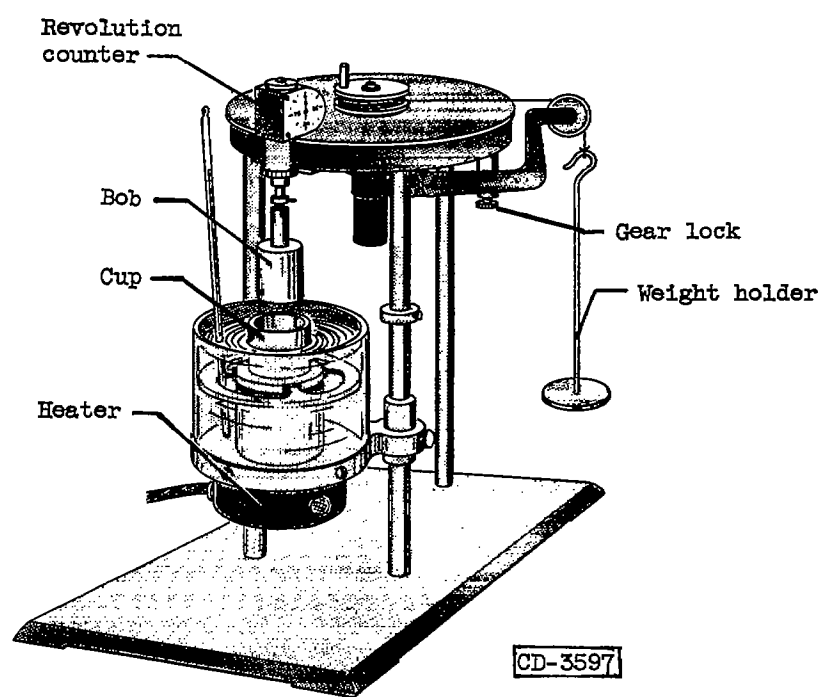
Figure 3. - Stormer rotational viscometer.

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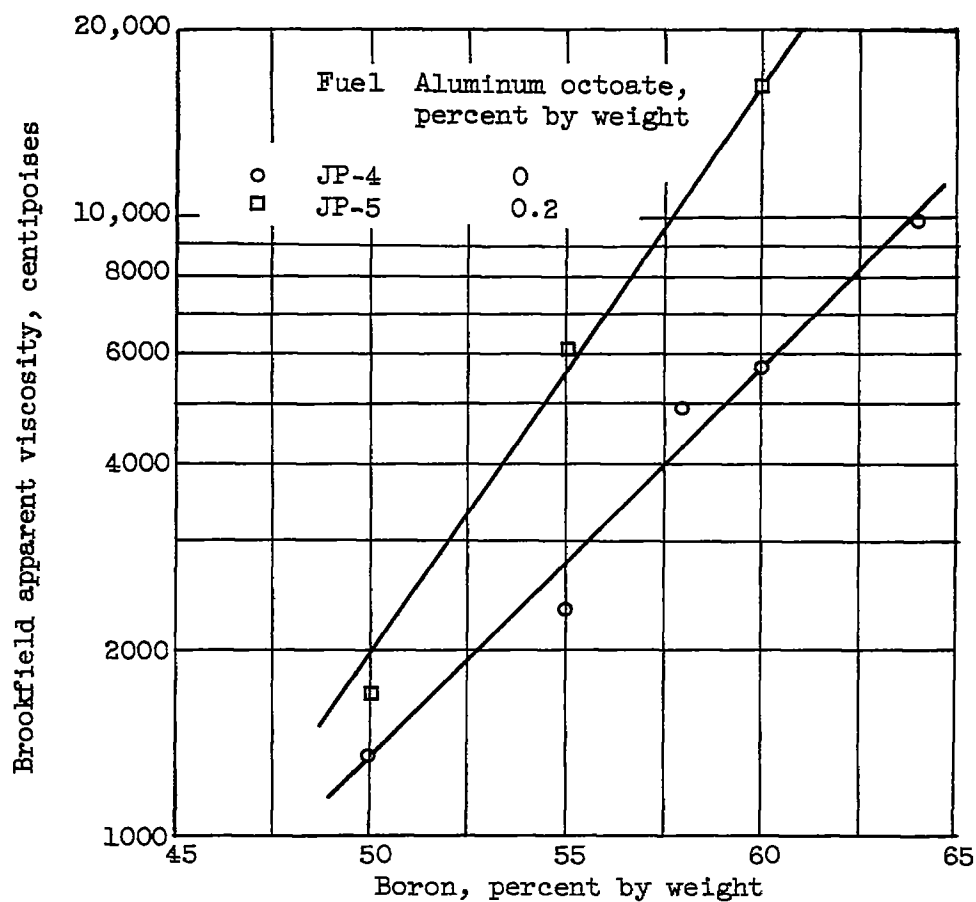
Figure 4. - Effect of boron concentration on Brookfield
apparent viscosity of slurries with and without
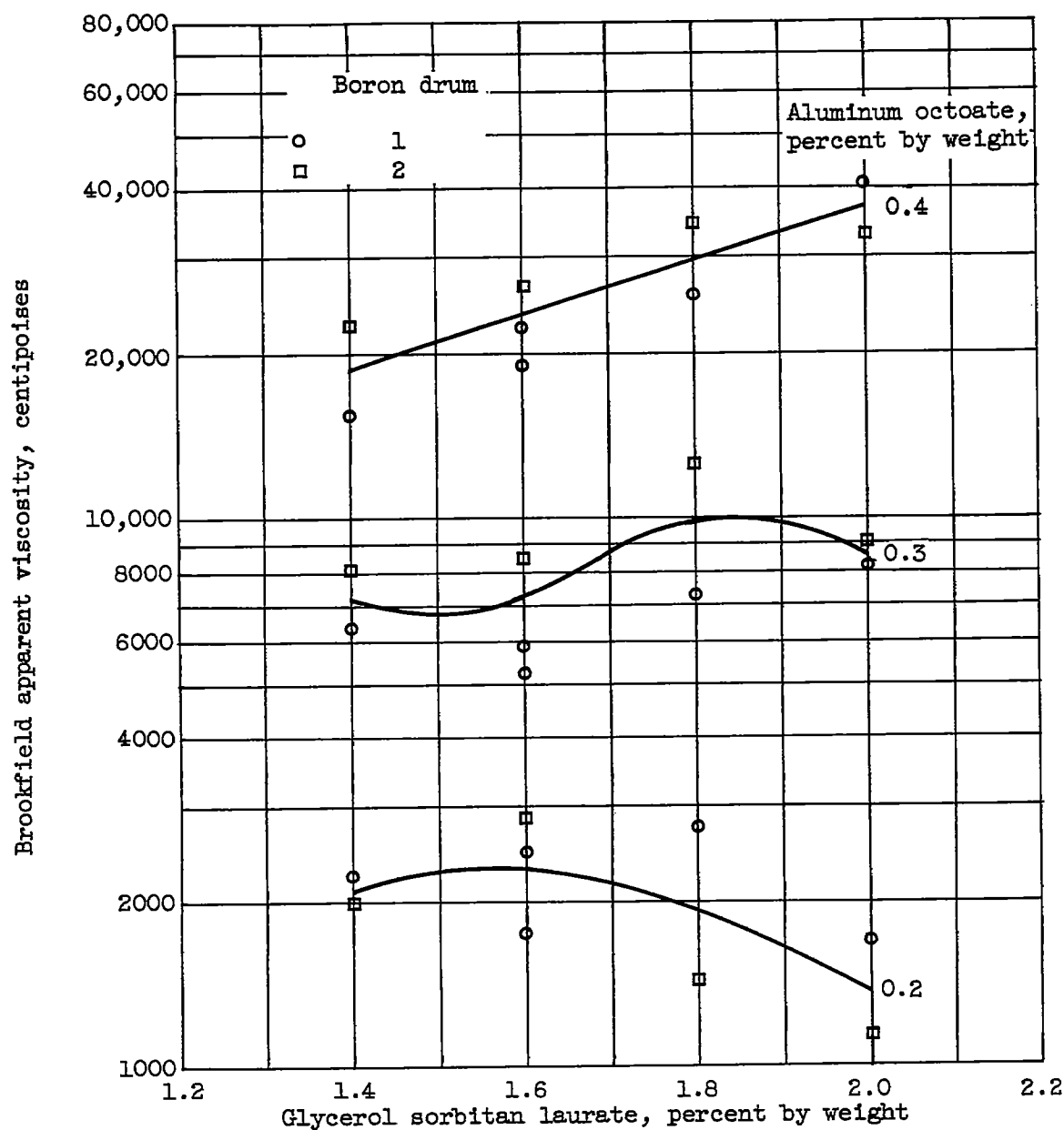aluminum octoate. Glycerol sorbitan laurate concen-
tration, 2.0 percent.

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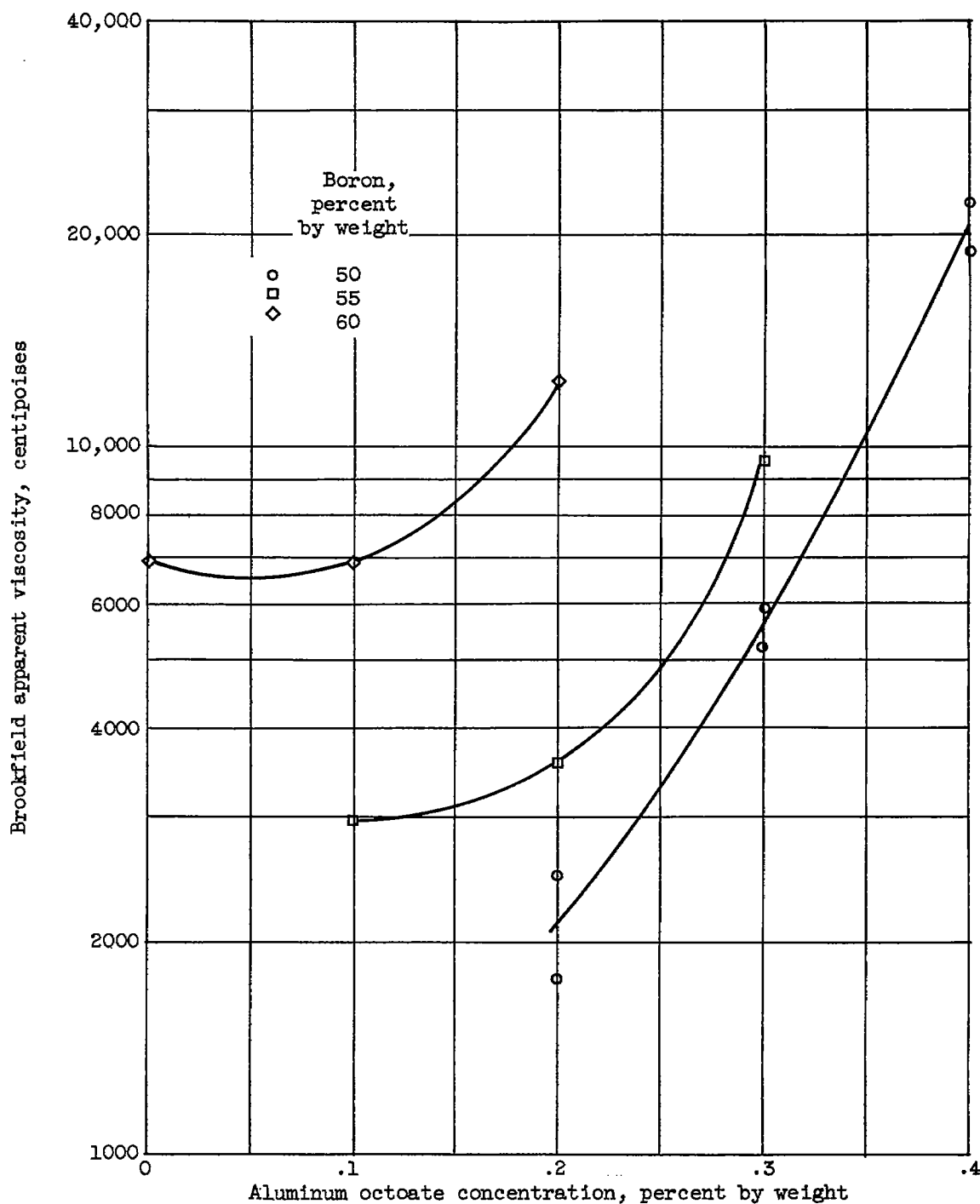
(a) Glycerol sorbitan laurate concentration, 1.6 percent.

Figure 5. - Effect of aluminum octoate concentration on Brookfield
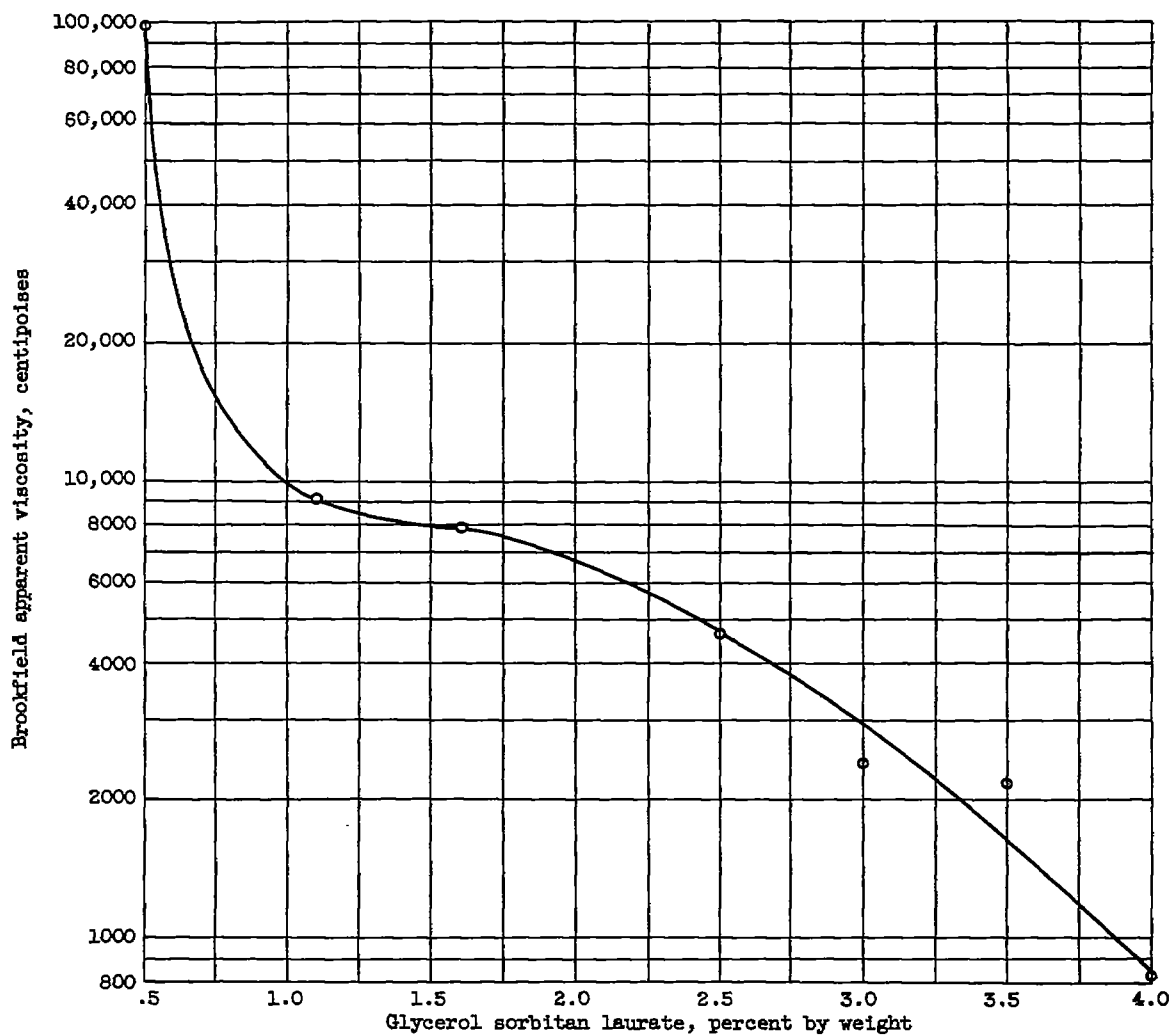apparent viscosities of boron slurries in JP-5 fuel.

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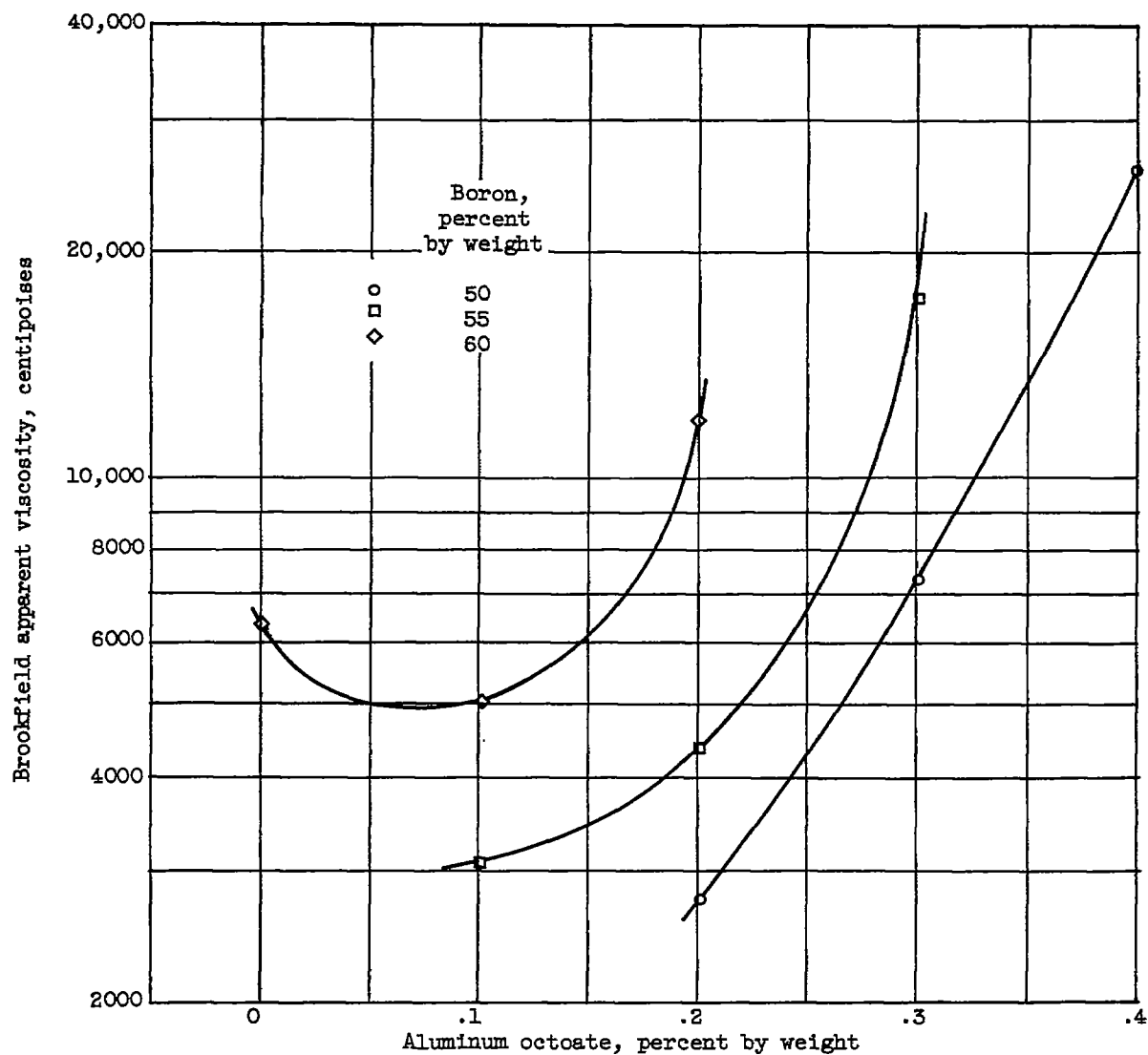
(b) Glycerol sorbitan laurate concentration, 1.8 percent.

Figure 5. - Continued. Effect of aluminum octoate concentration on Brookfield apparent viscosities of boron slurries in JP-5 fuel.

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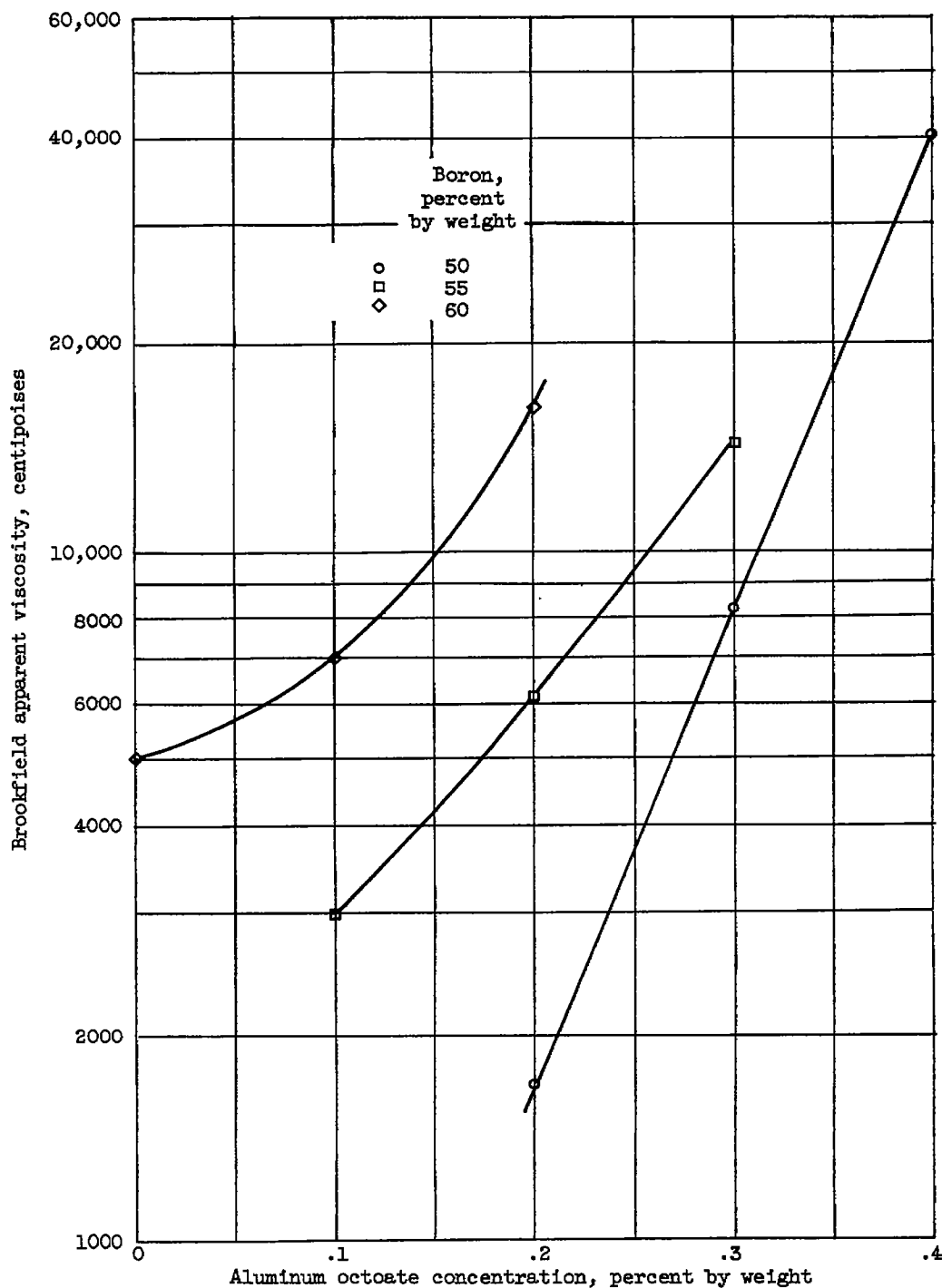
(c) Glycerol sorbitan laurate concentration, 2.0 percent.

Figure 5. - Concluded. Effect of aluminum octoate concentration on Brookfield apparent viscosities of boron slurries in JP-5 fuel.

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(a) Boron concentration, 50 percent.

Figure 6. - Effect of glycerol sorbitan laurate concentration on Brookfield apparent viscosities of boron slurries in JP-5 fuel.
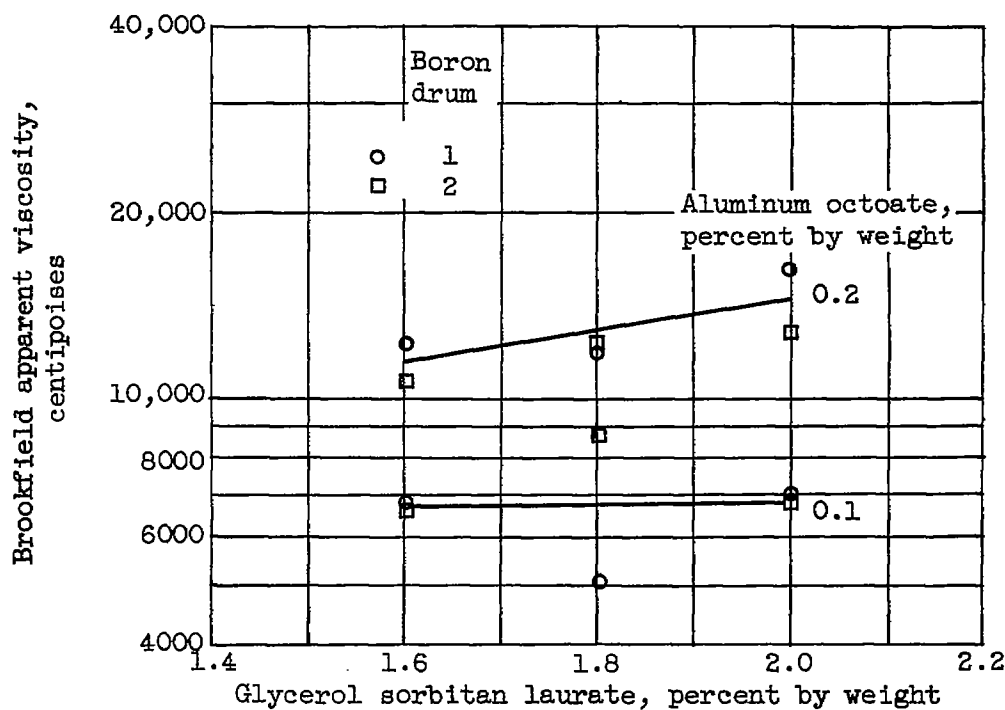
(b) Boron concentration, 60 percent.

Figure 6. - Concluded. Effect of glycerol sorbitan laurate concentration on Brookfield apparent viscosities of boron slurries in JP-5 fuel.

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Figure 7. - Effect of glycerol sorbitan laurate on Brookfield apparent viscosities of 50-percent-boron slurries in JP-5 fuel. Aluminum octoate concentration, 0.3 percent.
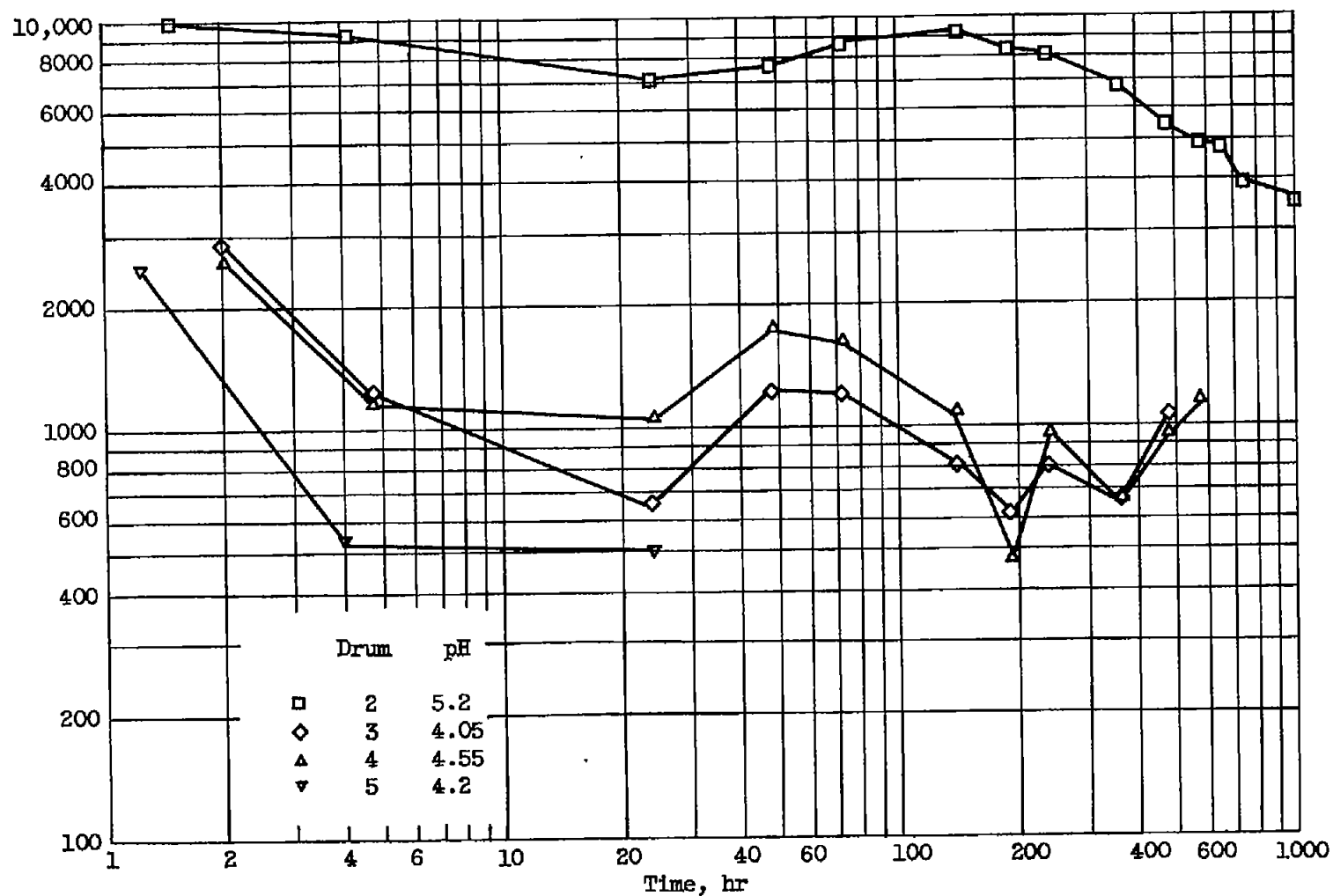
Figure 8. - Effect of powder acidity on viscosity and stability of 50-percent-boron slurries. Glycerol sorbitan laurate, 1.6 percent; aluminum octoate, 0.3 percent.
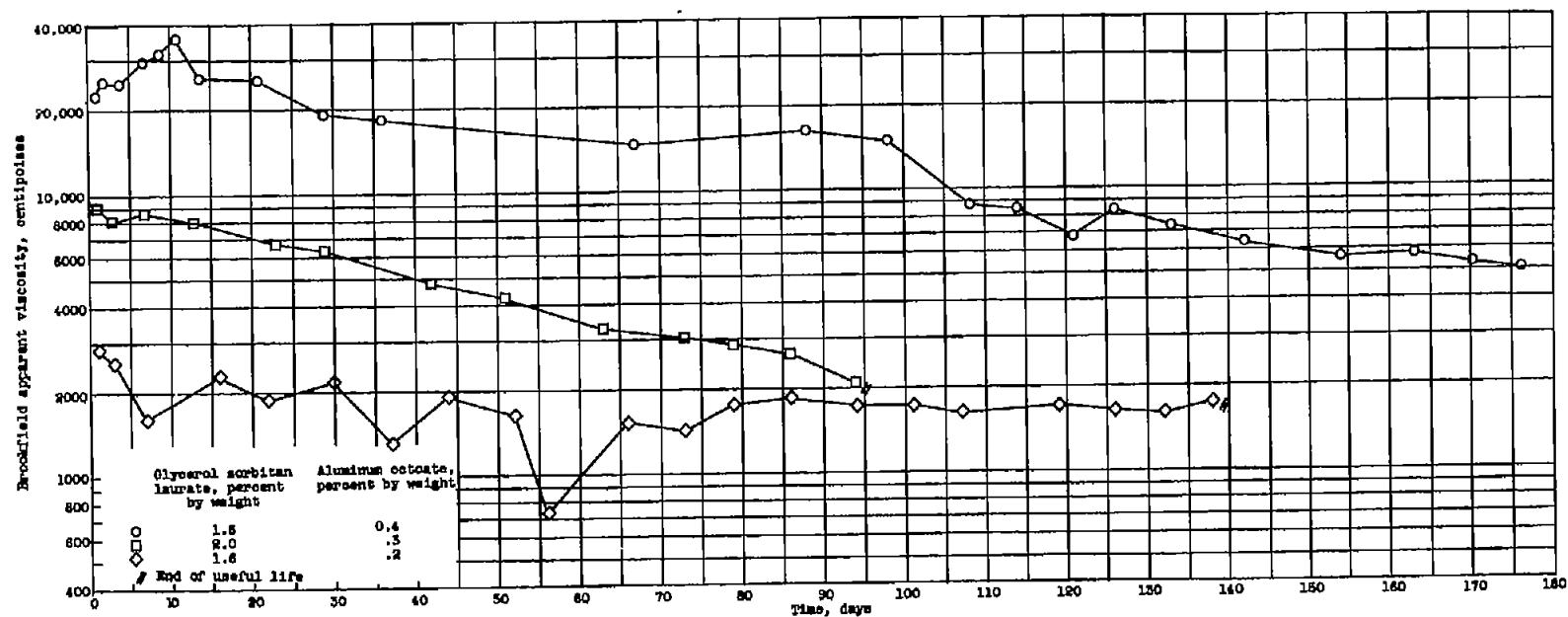
(a) Boron concentration, 50 percent.

Figure 9. - Effect of time on Brookfield apparent viscosity of boron slurries in JP-5 fuel.

(b) Boron concentration, 55 percent; glycerol sorbitan laurate concentration, 1.6 percent.
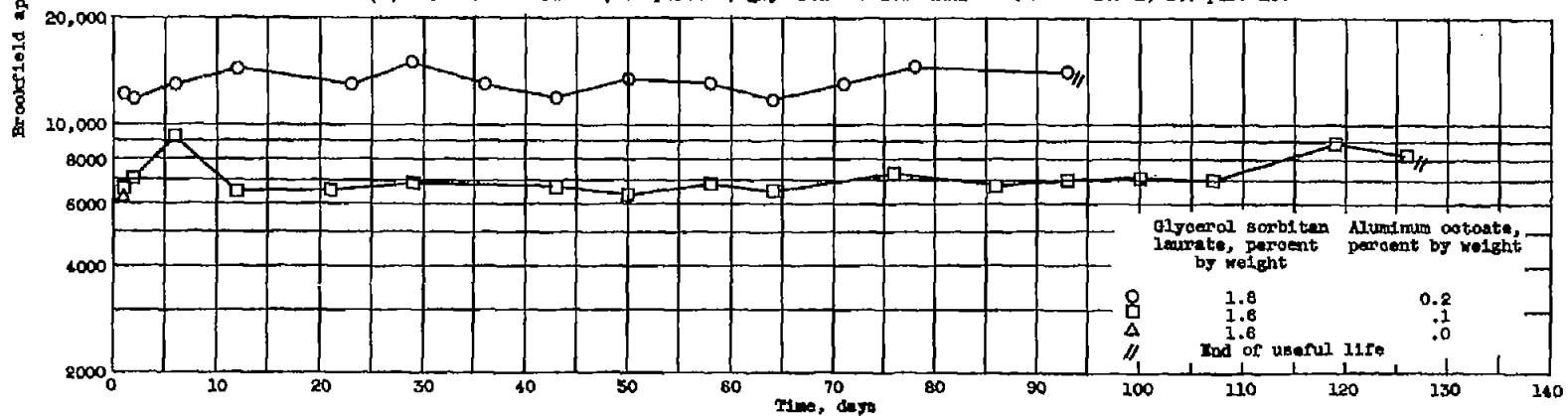
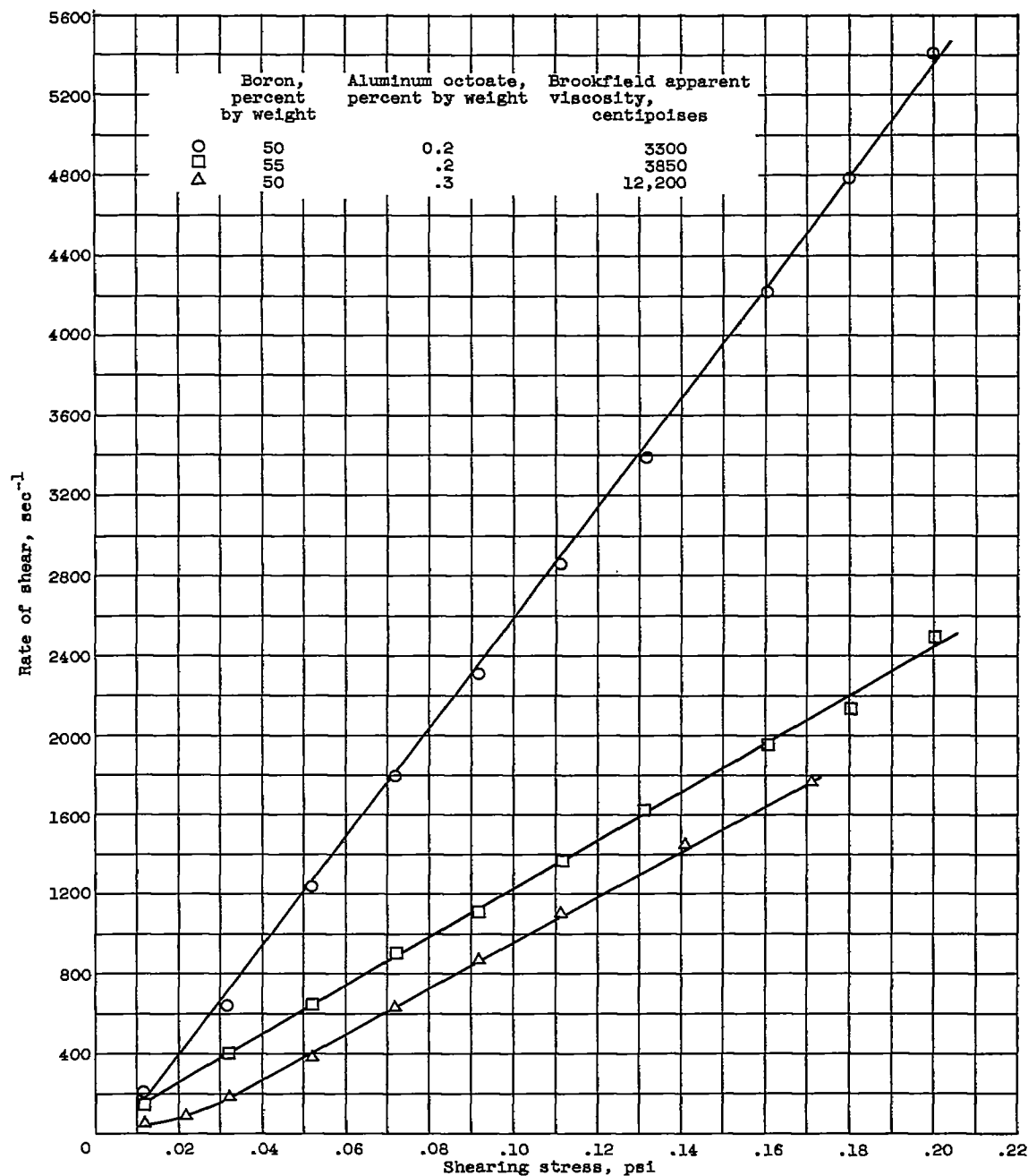(c) Boron concentration, 60 percent.

Figure 9. - Concluded. Effect of time on Brookfield apparent viscosity of boron slurries in JP-5 fuel.

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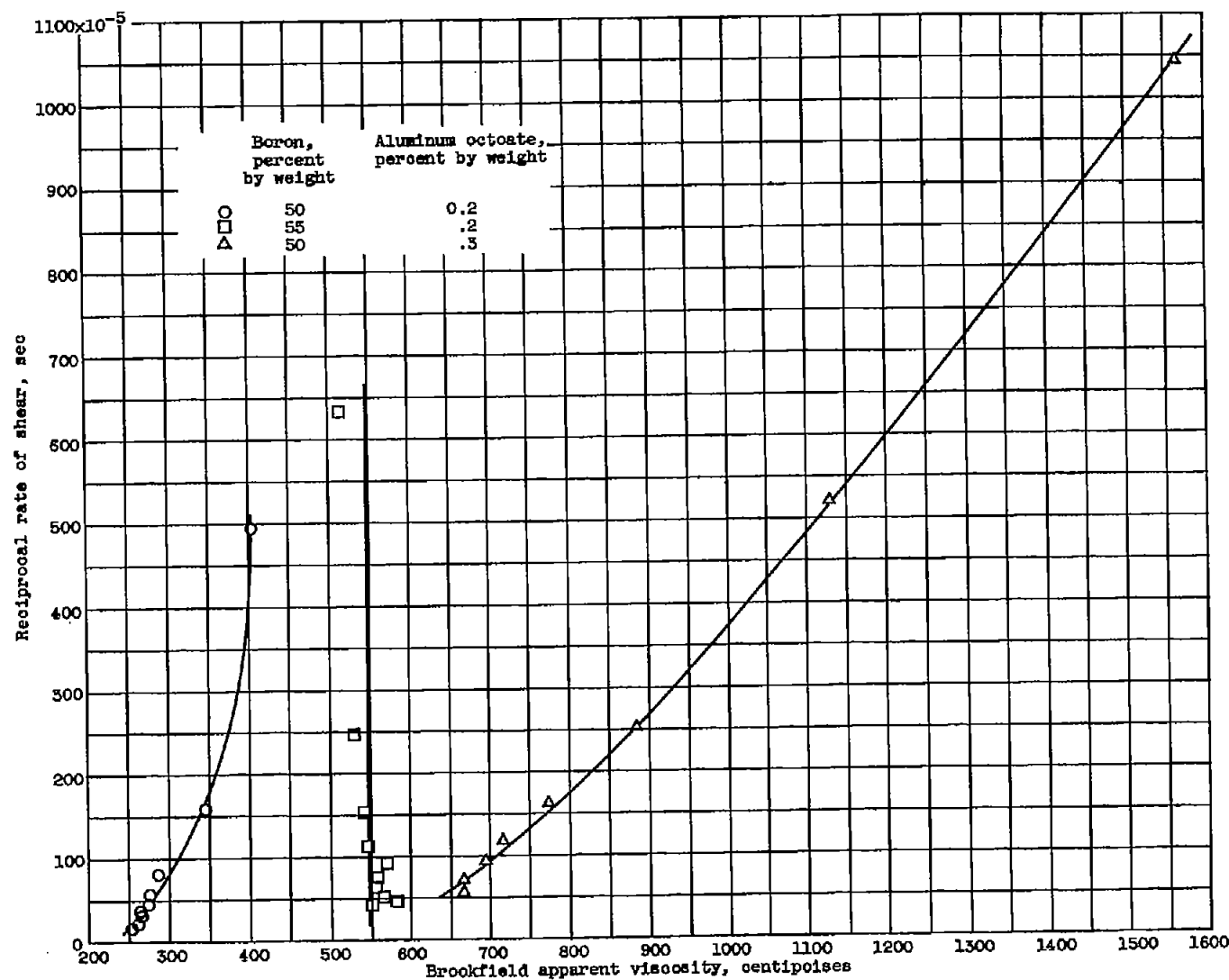|	Boron, percent by weight	Aluminum octoate, percent by weight	Brookfield apparent viscosity, centipoises
○	50	0.2	3300
□	55	.2	3850
△	50	.3	12,200

Figure 10. - Rate of shear against shearing stress for boron slurries in JP-5 fuel. Glycerol sorbitan laurate concentration, 1.6 percent.
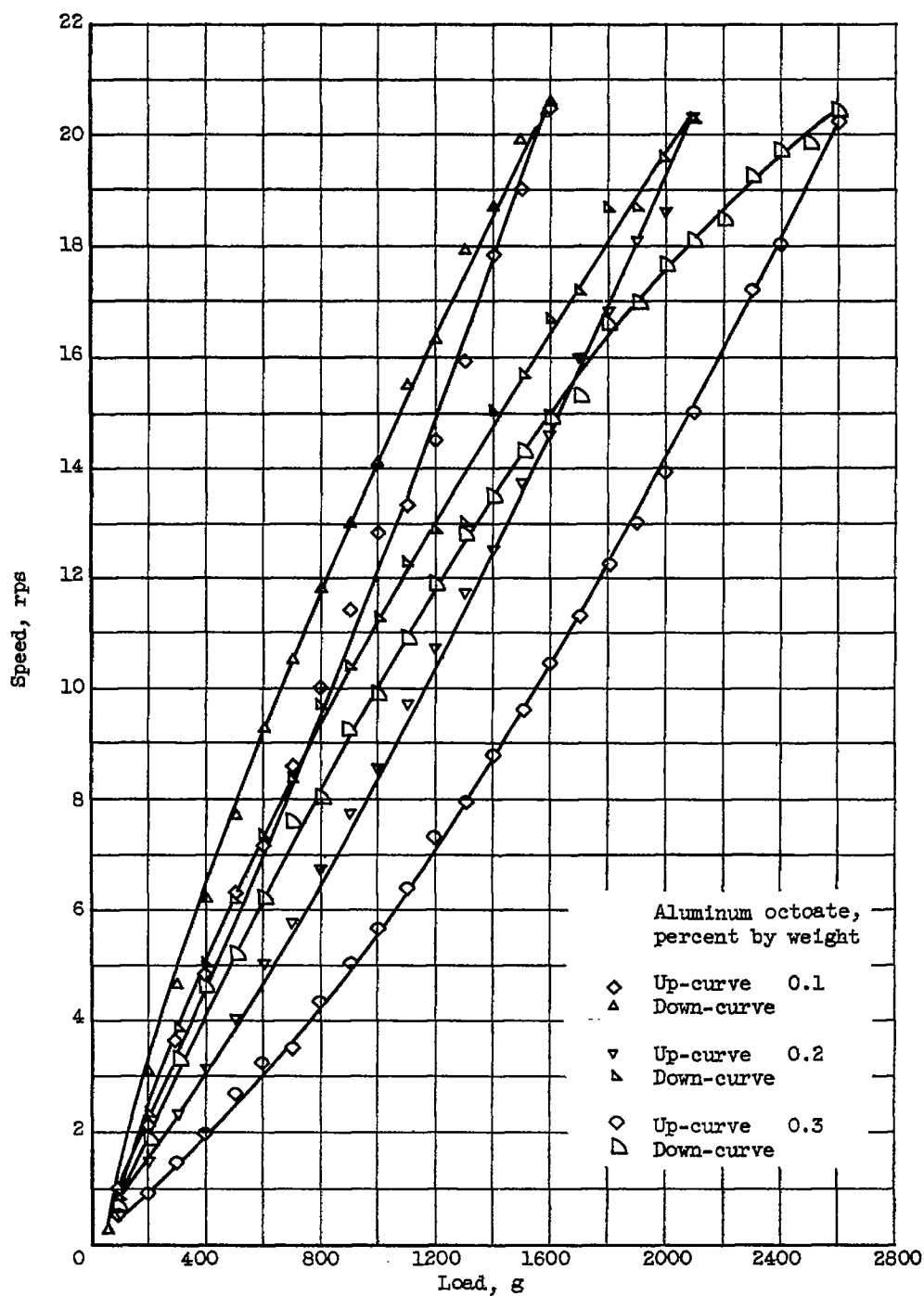
Figure 11. - Reciprocal of rate of shear against apparent viscosity for boron slurries in JP-5 fuel. Glycerol sorbitan laurate concentration, 1.6 percent.
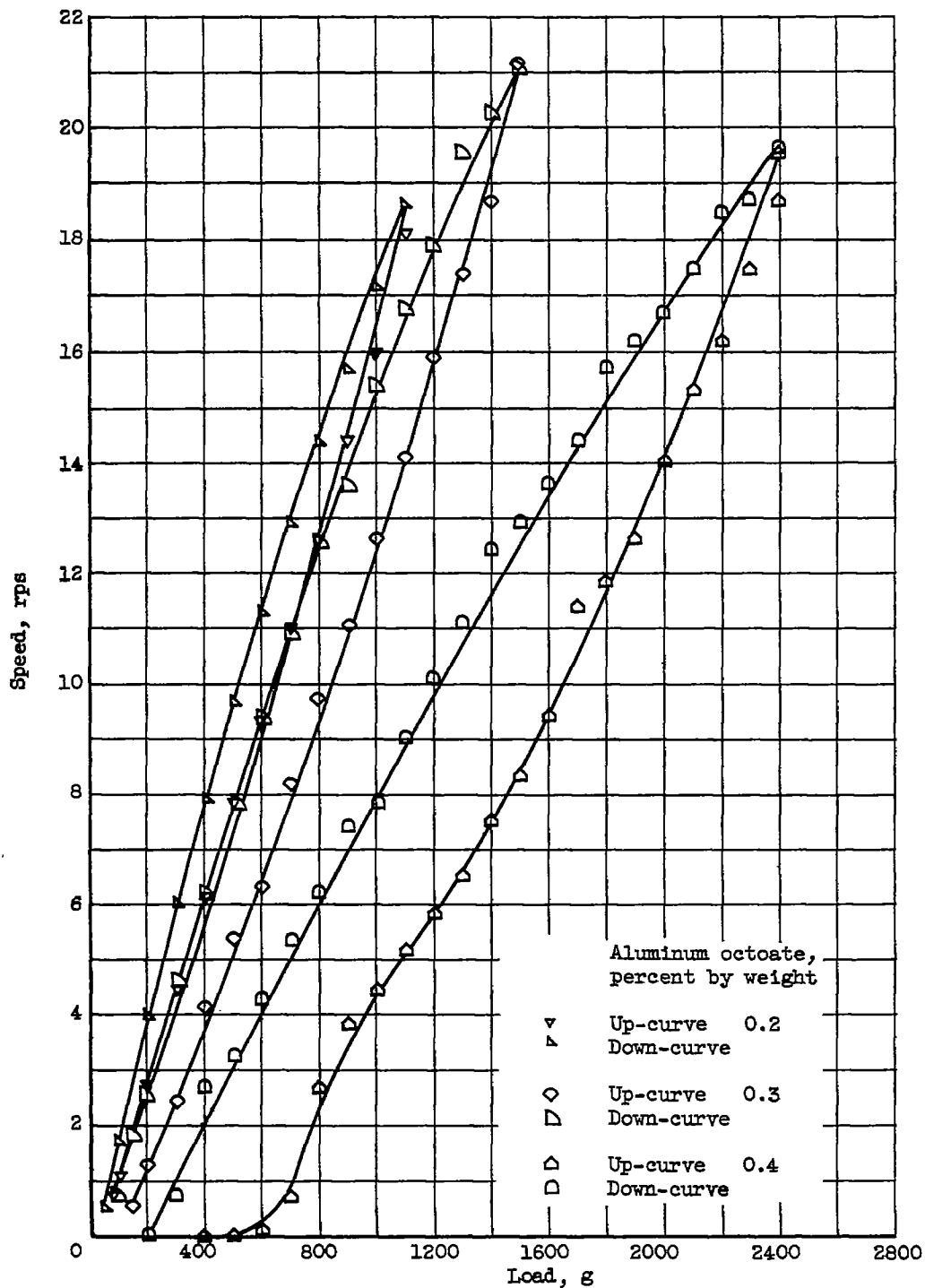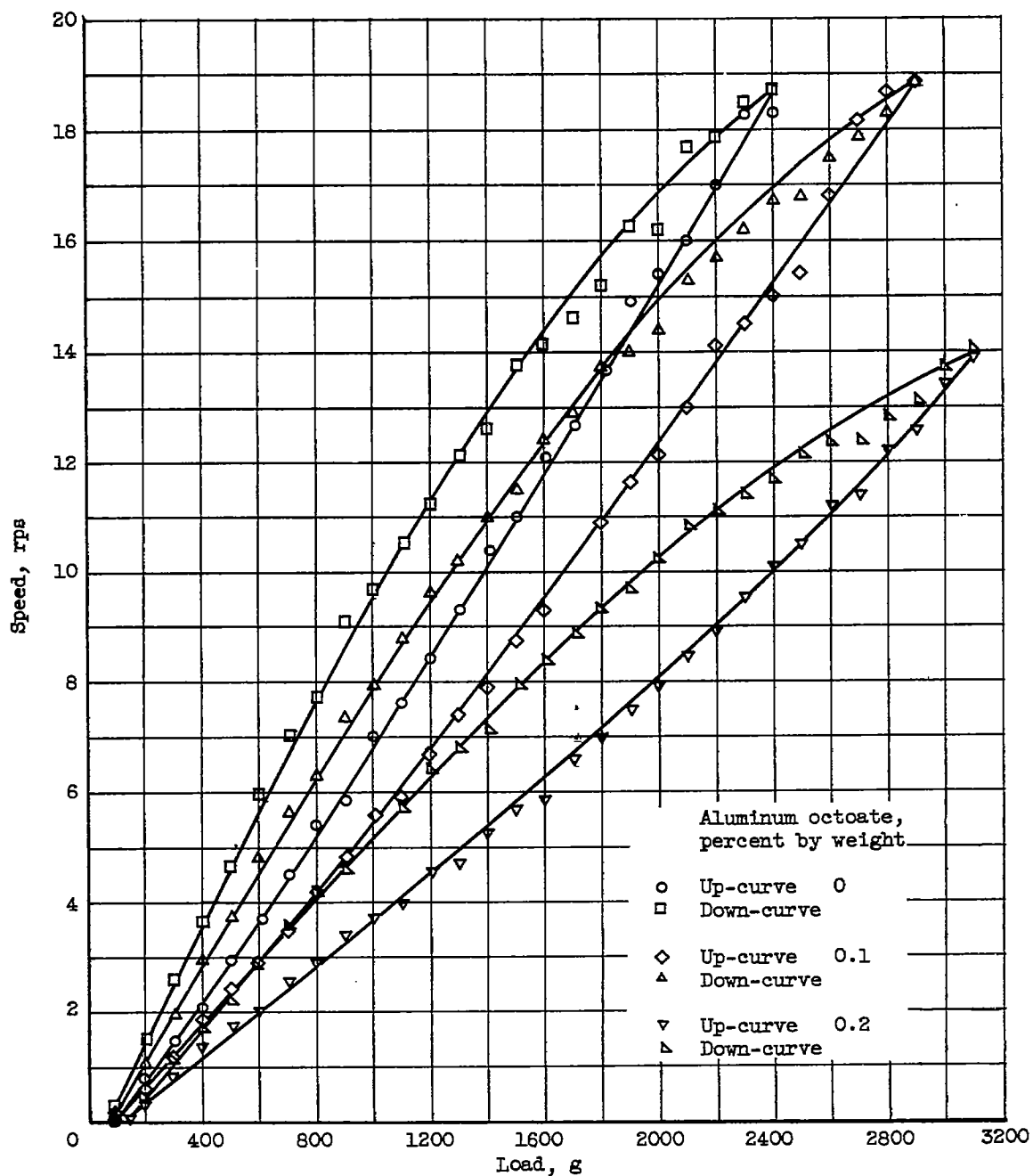
(a) Boron concentration, 50 percent; glycerol sorbitan laurate concentration, 2.0 percent.

Figure 12. - Stormer viscometer data for boron slurries in JP-5 fuel.

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(b) Boron concentration, 55 percent; glycerol sorbitan laurate
    concentration, 1.6 percent.

Figure 12. - Continued. Stormer viscometer data for boron slurries
    in JP-5 fuel.

(c) Boron concentration, 60 percent; glycerol sorbitan laurate concentration, 2.0 percent.

Figure 12. - Concluded. Stormer viscometer data for boron slurries in JP-5 fuel.